

U.S. DEPARTMENT OF ENERGY  
Federal Energy Technology Center

**REQUEST FOR PATENT CLEARANCE FOR  
RELEASE OF CONTRACTED RESEARCH DOCUMENTS**

TO: ~ For Technical Reports  
AAD Document Control  
U.S. Department of Energy - FETC  
MS 921-143  
P.O. Box 10940  
Pittsburgh, PA 15236-0940

Contract Agreement No. DE-AC21-94MC31258
Name & Phone No. of DOE COR Thomas P. Dorchak 304-285-4305

~ For Technical Papers/Journal Articles/Presentations  
Mark P. Dvorscak  
U.S. Department of Energy  
9800 S. Cass Avenue  
Argonne, IL 60439  
FAX: (630) 252-2779

**A. CONTRACTOR ACTION (CONTRACTOR COMPLETES PART A. 1-5)**

- 1. Document Title: Advanced Sulfur Control Concepts for Hot-Gas Desulfurization Technology
- 2. Type of Document: : Technical Progress Report ~ Topical Report ~ Final Technical Report  
~ Abstract ~ Technical Paper ~ Journal Article ~ Conference Presentation  
~ Other (please specify): \_\_\_\_\_
- 3. Date clearance needed: \_\_\_\_\_

4. Patent information.  
Yes No  
~ : Is any patentable subject matter disclosed in the report?  
~ ~ If so, has an invention disclosure been submitted to DOE Patent Counsel?  
If yes, identify disclosure number or DOE Case Number \_\_\_\_\_  
~ ~ Are there any patent-related objections to the release of this report? If so, state the  
objections.  
-  
-

5. Signed Original signed by Jeffrey W. Portzer Date May 3, 1999  
(Contractor)

Name & Phone No. Jeffrey W. Portzer (919) 541-8025  
Address Research Triangle Institute, P.O. Box 12194, Research Triangle Park, NC 27709-2194

**B. DOE PATENT COUNSEL ACTION**

- ~ Patent clearance for release of the above-identified document is granted.
- ~ Other: \_

Signed \_\_\_\_\_ Date \_\_\_\_\_  
(Patent Attorney)

Must be completed by the contractor.

DOE Contract No. DE-AC21-93MC31258--22  
RTI Project No. 93U-5869  
January 1, 1999 to March 31, 1999

**ADVANCED SULFUR CONTROL CONCEPTS  
FOR HOT GAS DESULFURIZATION TECHNOLOGY**

Quarterly Technical Progress Report

Submitted to

U.S. Department of Energy  
Federal Energy Technology Center  
3610 Collins Ferry Road  
P.O. Box 880  
Morgantown, WV 26507-0880

RTI Project No. 93U-5869

DOE Contract No. DE-AC21-94MC31258

January 1, 1999 to March 31, 1999

# **Advanced Sulfur Control Concepts for Hot Gas Desulfurization Technology**

Quarterly Technical Progress Report

Submitted to

U.S. Department of Energy  
Federal Energy Technology Center  
3610 Collins Ferry Road  
P.O. Box 880  
Morgantown, WV 26507-0880

Submitted by

Research Triangle Institute  
P.O. Box 12194  
Research Triangle Park, NC 27709

DOE COR: Thomas P. Dorchak  
RTI Project Manager: Santosh K. Gangwal  
RTI Project Engineer: Jeffrey W. Portzer

## TABLE OF CONTENTS

<b>Section</b>	<b>Page</b>
1.0 CONTRACT OBJECTIVE .....	1
2.0 TECHNICAL APPROACH .....	1
3.0 CONTRACT TASKS .....	1
4.0 OPEN ITEMS .....	3
5.0 PLANS FOR NEXT QUARTER .....	3

## LIST OF FIGURES

<b>Figure</b>	<b>Page</b>
1 Sufidation results from 27-cycle test of AHI-2 candidate sorbent . .	2

## **1. CONTRACT OBJECTIVE:**

The objective of this project is to develop a hot-gas desulfurization process scheme for control of H<sub>2</sub>S in HTHP coal gas that can be more simply and economically integrated with known regenerable sorbents in DOE/METC-sponsored work than current leading hot-gas desulfurization technologies. In addition to being more economical, the process scheme to be developed must yield an elemental sulfur byproduct.

## **2. TECHNICAL APPROACH:**

The Direct Sulfur Recovery Process (DSRP), a leading process for producing an elemental sulfur byproduct in hot-gas desulfurization systems, incurs a coal gas use penalty, because coal gas is required to reduce the SO<sub>2</sub> in regeneration off-gas to elemental sulfur. Alternative regeneration schemes, which avoid coal gas use and produce elemental sulfur, will be evaluated. These include (i) regeneration of sulfided sorbent using SO<sub>2</sub>; (ii) partial oxidation of sulfided sorbent in an O<sub>2</sub> starved environment; and (iii) regeneration of sulfided sorbent using steam to produce H<sub>2</sub>S followed by direct oxidation of H<sub>2</sub>S to elemental sulfur. Known regenerable sorbents will be modified to improve the feasibility of the above alternative regeneration approaches. Performance characteristics of the modified sorbents and processes will be obtained through lab- and bench-scale testing. Technical and economic evaluation of the most promising processes concept(s) will be carried out.

## **3. CONTRACT TASKS:**

### **Phase I - Concept Assessment:**

Completed.

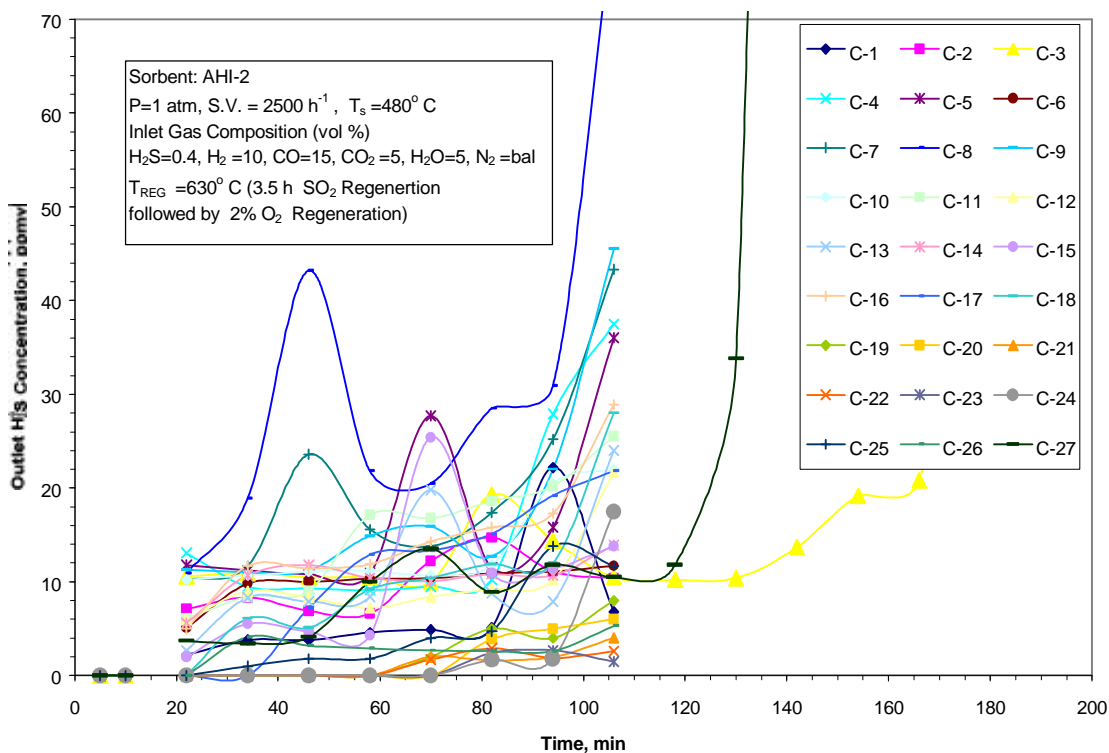
### **Phase II:**

#### Bench-Scale Sorbent Testing

The 27-cycle lab-scale test of a promising candidate sorbent, AHI-2, was completed. This test, conducted in cooperation with Hampton University, was carried out to qualify the sorbent for a high-pressure bench-scale test. The protocol for the cycles in the test series consisted of sulfidation at atmospheric pressure and 480 EC using a simulated coal gas containing 4000 ppm H<sub>2</sub>S. The sulfidation was followed by SO<sub>2</sub> regeneration for 3.5 h with 10% SO<sub>2</sub> in N<sub>2</sub> at 630 EC. The final step in each cycle was a polishing dilute air regeneration using 2% O<sub>2</sub> in N<sub>2</sub>. The sulfidation with H<sub>2</sub>S was preceded by 20 minutes of reductive regeneration with a sulfur-free simulated coal gas.

Figure 1 reports the results of the sulfidation runs. Excellent activity in terms of low outlet  $H_2S$  concentration was observed; concentrations below 20 ppmv were consistently obtained, with many runs below 10 ppmv. Interestingly, the later runs showed higher activity than the initial runs. Starting at cycle 19, concentrations of  $H_2S$  were undetectable (below 1 ppmv) during the first half of the sulfidation cycle. No  $H_2S$  or  $SO_2$  was detected during the reductive regeneration.

The  $SO_2$  regeneration consisted of 3.5 hours of 10%  $SO_2$  in nitrogen at 630 EC. There are no analytic data from this step. Elemental sulfur was formed but, due to the small scale of the apparatus, the yield was not quantified. The amount of regeneration accomplished with the  $SO_2$  was estimated by difference from the  $O_2$  regeneration data. Integration of the values for outlet  $SO_2$  concentration (obtained by GC) gave an estimate of the amount of residual sulfur in the sorbent that was regenerated by the dilute air stream. By these calculations, the  $SO_2$  regeneration resulted in up to 50% regeneration to elemental sulfur.



**Figure 1. Sulfidation results from 27-cycle test of AHI-2 candidate sorbent.**

The AHI series of sorbents is designed to be highly attrition-resistant. The data from a modified ASTM test (D 5757-95 protocol, except that a sample size smaller than specified

was used) bears this out. The attrition indices for AHI-1 and 2 were 0.5 and 1.2, respectively – similar to the values for the benchmark FCC catalysts. Based on these results, the AHI series of 1 and 2 were selected for scale-up and high pressure bench-scale testing.

### PSDF Field Test

Design, engineering, and construction are continuing on the renovation of the Mobile Laboratory for the Advanced Hot Gas Process (AHGP) field test. The data acquisition hardware and software (data logger) was installed and preliminary checkout completed. Modifications were started on the existing control panel to adapt it for process control of the renovated equipment skid. Temporary instrument air hookups were provided to stroke the valves. Plumbing and wiring errors were detected and corrected.

Orders were placed for several miscellaneous items for the process unit (valves, regulators, and another mass flow controller). Those additional hardware items (pressure regulators and mass flow controller) were received and installed.

### General

A paper is being prepared for presentation at the 4<sup>th</sup> International Symposium and Exhibition “Gas Cleaning at High Temperatures,” to be held in Karlsruhe, Germany, in September, 1999. It covers the laboratory-scale and bench-scale work that RTI has done to develop the SO<sub>2</sub> regeneration-based AHGP. Recent results from the lab-scale testing of new sorbent formulations at Hampton University are also included.

## **4. OPEN ITEMS**

The subcontract between RTI and Southern Company Services (SCS) at the PSDF is still not in place. That subcontract, part of the DSRP development contract (DE-AC21-93MC-30010) is required in order for SCS to install the coal gas slip stream line that will also be used for the field test portion of the AHGP project. If there is very much further delay in establishing the subcontract, the field test schedule will be negatively impacted.

## **5. PLANS FOR NEXT QUARTER:**

- ! Continue the construction activities in the Mobile Laboratory.
- ! Plan for scale-up and bench-scale testing (5 cycles each) of two candidate sorbents, AHI-1 and AHI-2, followed by a long duration test of one selected sorbent.

- ! Submit draft of Karlsruhe paper for review. Incorporate review comments and submit final version.